



PCT/EP200 4 / 0 0 1 5 1 2



INVESTOR IN PEOPLE

PRIORITY DOCUMENT
SUBMITTED OR TRANSMITTED IN
COMPLIANCE WITH
RULE 17.1(a) OR (b)

REC'D 22 MAR 2004

WIPO

PCT

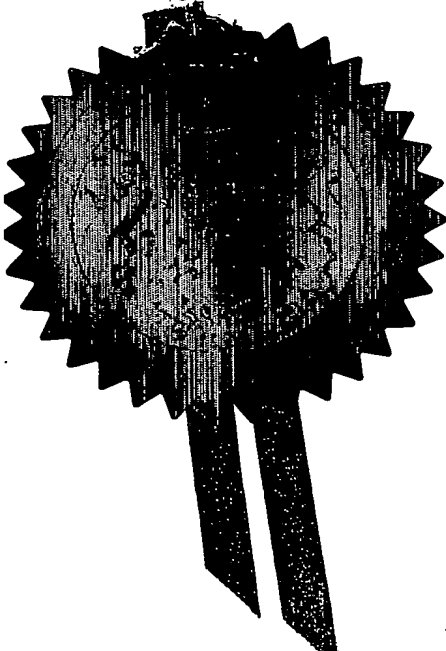
The Patent Office
Concept House
Cardiff Road
Newport
South Wales
NP10 8QQ

I, the undersigned, being an officer duly authorised in accordance with Section 74(1) and (4) of the Deregulation & Contracting Out Act 1994, to sign and issue certificates on behalf of the Comptroller-General, hereby certify that annexed hereto is a true copy of the documents as originally filed in connection with the patent application identified therein.

In accordance with the Patents (Companies Re-registration) Rules 1982, if a company named in this certificate and any accompanying documents has re-registered under the Companies Act 1980 with the same name as that with which it was registered immediately before re-registration save for the substitution as, or inclusion as, the last part of the name of the words "public limited company" or their equivalents in Welsh, references to the name of the company in this certificate and any accompanying documents shall be treated as references to the name with which it is so re-registered.

In accordance with the rules, the words "public limited company" may be replaced by p.l.c., plc, P.L.C. or PLC.

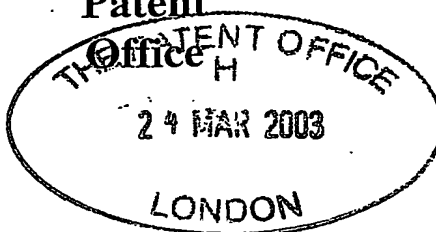
Re-registration under the Companies Act does not constitute a new legal entity but merely subjects the company to certain additional company law rules.



Signed

Dated 9 February 2004

BEST AVAILABLE COPY



The Patent Office

Cardiff Road
Newport
Gwent NP10 8QQ

Request for grant of a patent

(See the notes on the back of this form. You can also get an explanatory leaflet from the Patent Office to help you fill in this form)

1.	Your reference	C4296(C)/LH	
2.	Patent application number (The Patent Office will fill in this part)	0306790.7	24 MAR 2003
3.	Full name, address and postcode of the or of each applicant (<i>underline all surnames</i>)	UNILEVER PLC UNILEVER HOUSE, BLACKFRIARS LONDON, EC4P 4BQ	
	Patents ADP number (<i>if you know it</i>)	50426956002	
	If the applicant is a corporate body, give the country/state of its incorporation	UNITED KINGDOM	
4.	Title of the invention	DETERGENT COMPOSITION OR COMPONENT THEREFOR	
5.	Name of your agent (<i>if you have one</i>)	FRANSELLA Mary Evelyn Elliott, Peter William	
	"Address for Service" in the United Kingdom to which all correspondence should be sent (<i>including the postcode</i>)	PATENT DEPARTMENT, UNILEVER PLC COLWORTH HOUSE, SHARNBROOK BEDFORD, MK44 1LQ	
	Patents ADP number (<i>if you know it</i>)	6893440001	
6.	If you are declaring priority from one or more earlier patent applications, give the country and the date of filing of the or of each of these earlier applications and (<i>if you know it</i>) the or each application number	Country	Priority application number (<i>if you know it</i>)
			Date of filing (<i>day / month / year</i>)
7.	If this application is divided or otherwise derived from an earlier UK application, give the number and the filing date of the earlier application	Number of earlier application	Date of filing (<i>day/month/year</i>)
8.	Is a statement of inventorship and of right to grant of a patent required in support of this request? (<i>Answer 'Yes' if:</i> a) <i>any applicant named in part 3 is not an inventor, or</i> b) <i>there is an inventor who is not named as an applicant, or</i> c) <i>any named applicant is a corporate body.</i> <i>See note (d))</i>	YES	

F51/77 ES
30/6/03

Patents Form 1/77

9. Enter the number of sheets for any of the following items you are filing with this form. Do not count copies of the same document

Continuation sheets of this form

Description	14
Claim(s)	2 JUL
Abstract	1
Drawing(s)	

10. If you are also filing any of the following, state how many against each item.

Priority Documents

Translations of priority documents

Statement of inventorship and right to grant of a patent (Patents Form 7/77)


Request for preliminary examination and search (Patents Form 9/77) 1 ✓

Request for substantive examination (Patents Form 10/77)

Any other documents
(please specify)

11. I/We request the grant of a patent on the basis of this application.

Signature(s)



Date: 21/03/03

Sandra Jane EDWARDS, Authorised Signatory

12. Name and daytime telephone number of person to contact in the United Kingdom Louise Holland, Tel .01234 22 2082

Warning

After an application for a patent has been filed, the Comptroller of the Patent Office will consider whether publication or communication of the invention should be prohibited or restricted under Section 22 of the Patents Act 1977. You will be informed if it is necessary to prohibit or restrict your invention in this way. Furthermore, if you live in the United Kingdom, Section 23 of the Patents Act 1977 stops you from applying for a patent abroad without first getting written permission from the Patent Office unless an application has been filed at least 6 weeks beforehand in the United Kingdom for a patent for the same invention and either no direction prohibiting publication or communication has been given, or any such direction has been revoked.

Notes

- If you need help to fill in this form or you have any questions, please contact the Patent Office on 0645 500505.
- Write your answers in capital letters using black ink or you may type them.
- If there is not enough space for all the relevant details on any part of this form, please continue on a separate sheet of paper and write "see continuation sheet" in the relevant part(s). Any continuation sheet should be attached to this form.
- If you have answered 'Yes' Patents Form 7/77 will need to be filed.
- Once you have filled in the form you must remember to sign and date it.
- For details of the fee and ways to pay please contact the Patent Office.

- 1 -

DETERGENT COMPOSITION OR COMPONENT THEREFORTECHNICAL FIELD

5 The present invention relates to a particulate detergent composition or component therefor which comprises a bi- or tri- valent metal ion carboxylic acid salt.

BACKGROUND AND PRIOR ART

10

Modern particulate detergent composition contain a wide variety of ingredients. At the same time the consumer requires a particulate detergent composition which has acceptable physical characteristics such as, for example,

15 being flowable and pourable in the case of powders and having good dispensing and dissolution properties in the case of tablets. The physical properties must remain acceptable even after storage for a number of weeks in the supply chain.

20

Many ingredients which provide a useful benefit to the detergent composition are difficult to include because they are liquid. However, liquid ingredients are not in themselves a problem as long as sufficient liquid carrying capacity is present in the solid portion of the detergent.

25 Of more difficulty are the ingredients which are hygroscopic, which may be either liquid or solid.

Hygroscopic materials are difficult to handle since they absorb atmospheric moisture over time during storage and can

30 lead to a deleterious effect on physical properties such as undesired caking and subsequent reduction in flowability at

the point of use or a reduction is dispensability,
dispersability or dissolution rate.

5 US 4,196,095 (Church & Dwight) discloses a carbonate based
detergent composition which comprises magnesium stearate in
order to reduce insoluble lump formation in cold water.

10 The present inventors have found that specific carboxylic
acid salts are especially effective at reducing the negative
effects on physical properties of particulate detergent
compositions or components therefor due to moisture uptake.

DETAILED DESCRIPTION OF INVENTION

15 The bi- or tri- valent metal ion carboxylic acid salt

The carboxylic acid salt preferably has a metal ion selected
from magnesium, calcium, zinc, aluminium, lead, iron or
mixtures thereof, preferably magnesium.

20 Preferably the carboxylic acid salt has from 10 to 22,
preferably from 14 to 20 carbon atoms, more preferably 18
carbon atoms. Most preferably the salt is magnesium
stearate having 18 carbon atoms.

25 The carboxylic acid salt is preferably in particulate form.
If so it preferably has a number average particle size of
from 0.1 to 500 micrometres, preferably from 1 to 200
micrometres, more preferably from 2 to 100 micrometres, most
30 preferably from 3 to 50 micrometres, or even from 3 to
20 micrometres.

The carboxylic acid salt may be present substantially homogeneously throughout the composition or component.

Alternatively the carboxylic acid salt may be present at the surface of the particles at a higher concentration than the average concentration in the composition or component. In a preferred embodiment the carboxylic acid salt is present on the surface of the particles, and this is often referred to in the art as layering.

10 The carboxylic acid salt is preferably present at a level of from 0.1 to 10 wt%, preferably from 1 to 8 wt%, more preferably from greater than 2 to 6 wt%, or even from 2.5 to 5 wt%, based on the weight of the composition or component before addition of the carboxylic acid salt.

15

The carboxylic acid salt may simply be added to a previously manufactured particulate detergent composition or component. Preferably the carboxylic acid salt is particulate and is layered onto the surface of the detergent composition or component.

20

The Composition or Component Therefor

The present invention applies equally to detergent compositions and to components of detergent compositions which are designed to be added to an already manufactured detergent composition. For example the invention applies to detergent base powder particles made by spray-drying or granulation as well as adjunct components such as bleach or enzyme granules, perfume granules, effervescent granules

30

etc. Preferred perfume granules comprise maltose and Polybutylmethacrylate.

5 The term "hygroscopicity value", as used herein, means the level of moisture uptake by the composition or component, as measured by the percent increase in weight of the particles under the following test method. The hygroscopicity value is determined by placing 2 grams of particles (approximately 10 500 microns in size) in an open petri dish under conditions of 37°C and 70% relative humidity for a period of 4 weeks. The percent increase in weight of the particles at the end of this time is the particles' hygroscopicity value as used herein.

15 Since the carboxylic acid salt reduces the negative effects of moisture uptake, the invention relates to compositions or components which are hygroscopic. It is not known whether the carboxylic acid salt reduces the amount of water uptake or whether it prevents caking without reducing water uptake. 20 In any case the invention applies to detergent compositions or components which, without carboxylic acid salt being present, have a hygroscopicity value of at least 2%, preferably greater than 5%, preferably greater than 10%, more preferably greater than 20%, or even greater than 30%, 25 or even greater than 40% or even greater than 50%.

Organic Detergent Surfactant

Finished detergent compositions of the present invention

~~30 contain an organic detergent surfactant. Components for~~

detergent compositions which are also part of the invention do not necessarily comprise surfactant.

Detergent-active compounds (surfactants) may be chosen from soap and non-soap anionic, cationic, nonionic, amphoteric and zwitterionic detergent-active compounds, and mixtures thereof. Many suitable detergent-active compounds are available and are fully described in the literature, for example, in "Surface-Active Agents and Detergents", Volumes I and II, by Schwartz, Perry and Berch. The preferred detergent-active compounds that can be used are soaps and synthetic non-soap anionic and nonionic compounds. The total amount of surfactant present is suitably within the range of from 5 to 60 wt%, preferably from 5 to 40 wt%.

Anionic surfactants are well-known to those skilled in the art. Examples include alkylbenzene sulphonates, particularly linear alkylbenzene sulphonates having an alkyl chain length of C₈-C₁₅; primary and secondary alkylsulphates, particularly C₈-C₂₀ primary alkyl sulphates; alkyl ether sulphates; olefin sulphonates; alkyl xylene sulphonates; dialkyl sulphosuccinates; and fatty acid ester sulphonates. Sodium salts are generally preferred. Nonionic surfactants that may be used include the primary and secondary alcohol ethoxylates, especially the C₈-C₂₀ aliphatic alcohols ethoxylated with an average of from 1 to 20 moles of ethylene oxide per mole of alcohol, and more especially the C₁₀-C₁₅ primary and secondary aliphatic alcohols ethoxylated with an average of from 1 to 10 moles of ethylene oxide per mole of alcohol. Non-ethoxylated

nonionic surfactants include alkanolamides, alkylpolyglycosides, glycerol monoethers, and polyhydroxyamides (glucamide).

- 5 Cationic surfactants that may be used include quaternary ammonium salts of the general formula $R_1R_2R_3R_4N^+ X^-$ wherein the R groups are long or short hydrocarbyl chains, typically alkyl, hydroxyalkyl or ethoxylated alkyl groups, and X is a solubilising anion (for example, compounds in which R_1 is a
- 10 C_8 - C_{22} alkyl group, preferably a C_8 - C_{10} or C_{12} - C_{14} alkyl group, R_2 is a methyl group, and R_3 and R_4 , which may be the same or different, are methyl or hydroxyethyl groups); and cationic esters (for example, choline esters).
- 15 Amphoteric and zwitterionic surfactants that may be used include alkyl amine oxides, betaines and sulphobetaines. In accordance with the present invention, the detergent surfactant (a) most preferably comprises an anionic
- 20 sulphonate or sulphate surfactant optionally in admixture with one or more cosurfactants selected from ethoxylated nonionic surfactants, non-ethoxylated nonionic surfactants, ethoxylated sulphate anionic surfactants, cationic surfactants, amine oxides, alkanolamides and combinations thereof.
- 25 Surfactants are preferably present in a total amount of from 5 to 60 wt%, more preferably from 10 to 40 wt%.
-

Detergency Builder

Finished detergent compositions of the present invention preferably contain a detergency builder, although it is conceivable that formulations without any builder are possible.

The compositions of the invention suitably contain from 10 to 80%, preferably from 15 to 70% by weight, of detergency builder. Preferably, the quantity of builder is in the range of from 15 to 50% by weight.

Preferably the builder is selected from zeolite, sodium tripolyphosphate, sodium carbonate, sodium citrate, layered silicate, and combinations of these.

The zeolite used as a builder may be the commercially available zeolite A (zeolite 4A) now widely used in laundry detergent powders. Alternatively, the zeolite may be maximum aluminium zeolite P (zeolite MAP) as described and claimed in EP 384 070B (Unilever), and commercially available as Doucil (Trade Mark) A24 from Ineos Silicas Ltd, UK.

Zeolite MAP is defined as an alkali metal aluminosilicate of zeolite P type having a silicon to aluminium ratio not exceeding 1.33, preferably within the range of from 0.90 to 1.33, preferably within the range of from 0.90 to 1.20. Especially preferred is zeolite MAP having a silicon to aluminium ratio not exceeding 1.07, more preferably about 1.00. The particle size of the zeolite is not critical.

Zeolite A or zeolite MAP of any suitable particle size may be used.

Also preferred according to the present invention are

- 5 phosphate builders, especially sodium tripolyphosphate. This may be used in combination with sodium orthophosphate, and/or sodium pyrophosphate.

- 10 Other inorganic builders that may be present additionally or alternatively include sodium carbonate, layered silicate, amorphous aluminosilicates.

- Organic builders that may be present include polycarboxylate polymers such as polyacrylates and acrylic/maleic
15 copolymers; polyaspartates; monomeric polycarboxylates such as citrates, gluconates, oxydisuccinates, glycerol mono-di- and trisuccinates, carboxymethyloxysuccinates, carboxy-methyloxymalonates, dipicolinates, hydroxyethyl-
iminodiacetates, alkyl- and alkenylmalonates and succinates;
20 and sulphonated fatty acid salts.

Organic builders may be used in minor amounts as supplements to inorganic builders such as phosphates and zeolites.

- Especially preferred supplementary organic builders are
25 citrates, suitably used in amounts of from 5 to 30 wt %, preferably from 10 to 25 wt %; and acrylic polymers, more especially acrylic/maleic copolymers, suitably used in amounts of from 0.5 to 15 wt %, preferably from 1 to 10 wt%. Builders, both inorganic and organic, are preferably present

~~30 in alkali metal salt, especially sodium salt, form.~~

Other Detergent Ingredients

As well as the surfactants and builders discussed above, the compositions may optionally contain bleaching components and
5 other active ingredients to enhance performance and properties.

These optional ingredients may include, but are not limited to, any one or more of the following: soap, peroxyacid and
10 persalt bleaches, bleach activators, sequestrants, cellulose ethers and esters, other antiredeposition agents, sodium sulphate, sodium silicate, sodium chloride, calcium chloride, sodium bicarbonate, other inorganic salts, proteases, lipases, cellulases, amylases, other detergent
15 enzymes, fluorescers, photobleaches, polyvinyl pyrrolidone, other dye transfer inhibiting polymers, foam controllers, foam boosters, acrylic and acrylic/maleic polymers, citric acid, soil release polymers, fabric conditioning compounds, coloured speckles, and perfume.

20

Detergent compositions according to the invention may suitably contain a bleach system. The bleach system is preferably based on peroxy bleach compounds, for example, inorganic persalts or organic peroxyacids, capable of
25 yielding hydrogen peroxide in aqueous solution. Suitable peroxy bleach compounds include organic peroxides such as urea peroxide, and inorganic persalts such as the alkali metal perborates, percarbonates, perphosphates, persilicates and persulphates. Preferred inorganic persalts are sodium
30 perborate monohydrate and tetrahydrate, and sodium percarbonate. Especially preferred is sodium percarbonate

having a protective coating against destabilisation by moisture. Sodium percarbonate having a protective coating comprising sodium metaborate and sodium silicate is disclosed in GB 2 123 044B (Kao).

5

The peroxy bleach compound is suitably present in an amount of from 5 to 35 wt%, preferably from 10 to 25 wt%.

10 The peroxy bleach compound may be used in conjunction with a bleach activator (bleach precursor) to improve bleaching action at low wash temperatures. The bleach precursor is suitably present in an amount of from 1 to 8 wt%, preferably from 2 to 5 wt%.

15 Preferred bleach precursors are peroxycarboxylic acid precursors, more especially peracetic acid precursors and peroxybenzoic acid precursors; and peroxycarbonic acid precursors. An especially preferred bleach precursor
20 suitable for use in the present invention is N,N,N',N'-tetracetyl ethylenediamine (TAED). Also of interest are peroxybenzoic acid precursors, in particular, N,N,N-trimethylammonium toluoyloxy benzene sulphonate.

25 A bleach stabiliser (heavy metal sequestrant) may also be present. Suitable bleach stabilisers include ethylenediamine tetraacetate (EDTA) and the polyphosphonates such as Dequest (Trade Mark), EDTMP.

Although, as previously indicated, in one preferred

~~30 embodiment of the invention enzymes are preferably absent,~~
in other embodiments detergent enzymes may be present.

Suitable enzymes include the proteases, amylases, cellulases, oxidases, peroxidases and lipases usable for incorporation in detergent compositions.

- 5 In particulate detergent compositions, detergency enzymes are commonly employed in granular form in amounts of from about 0.1 to about 3.0 wt%. However, any suitable physical form of enzyme may be used in any effective amount.
- 10 Antiredeposition agents, for example cellulose esters and ethers, for example sodium carboxymethyl cellulose, may also be present.

- The compositions may also contain soil release polymers, for example sulphonated and unsulphonated PET/POET polymers, both end-capped and non-end-capped, and polyethylene glycol/polyvinyl alcohol graft copolymers such as Sokolan (Trade Mark) HP22. Especially preferred soil release polymers are the sulphonated non-end-capped polyesters described and claimed in WO 95 32997A (Rhodia Chimie).
- 15
- 20

Product Form and Preparation

- Powder detergent composition of low to moderate bulk density may be prepared by spray-drying a slurry, and optionally postdosing (dry-mixing) further ingredients.
- 25

"Concentrated" or "compact" powders may be prepared by mixing and granulating processes, for example, using a high-speed mixer/granulator, or other non-tower processes.

Tablets may be prepared by compacting powders, especially "concentrated" powders.

EXAMPLES

5

Perfume granules were made up to the following formulation using the process described in US 6,024,943:

Ingredient	Wt%
Polybutylmethacrylate beads	24
Perfume	24
Maltose	50
Water	2

10

Particulate layering agent was then added to the perfume granule. For comparative example A, no layering agent was added. 20 g of each sample of the granules were then put on open uncontrolled storage under estimated conditions of 20°C

15

and 40-45%RH. The results are given in the table below.

Example	Layering agent	wt%	Storage time			
			6 hours	3 days	6 days	8 weeks
A	None	-	D	d	-	-
B	Zeolite A24 ⁽¹⁾	2%	A	b	d	-
C	Zeolite A24 ⁽¹⁾	3%	A	b	d	-
D	Zeolite A24 ⁽¹⁾	4%	A	b	d	-
E	Zeolite A24 ⁽¹⁾	5%	A	a	c	-
F	CaCO ₃ ⁽²⁾	2%	A	b	d	-
G	CaCO ₃ ⁽²⁾	5%	A	a	d	-
1	Mg-stearate ⁽³⁾	2%	A	b	b	-
2	Mg-stearate ⁽³⁾	3%	A	a	a	a
3	Mg-stearate ⁽³⁾	4%	A	a	a	a
4	Mg-stearate ⁽³⁾	5%	A	a	a	a
H	Di calcium phosphate ⁽⁴⁾	2%	B	d	-	-
I	Di calcium phosphate ⁽⁴⁾	5%	a	d	-	-

a No caking/flows well

b Light caking/flows less well

5 c Caking

d Heavy caking

(1) Doucil A24, supplied by Ineos Silicas

(2) Supplied by JT Baker

(3) Supplied by Acros Chemical with an $d_{3,2}$ particle size of 7.3 micrometres as measured on a Malvern Mastersizer.

(4) Supplied by Acros Chemical

5

In a second series of experiments, magnesium stearate was compared to sodium stearate. In these examples, 50 g of the perfume granules were layered and put onto open storage under conditions of 37°C/70%RH.

10

The following results were obtained:

Example	Layering agent	wt%	Storage time			
			15 hours	24 hours	2 days	5 days
S	Mg-stearate ⁽³⁾	3%	a	a	A	a
J	Na-stearate ⁽⁵⁾	3%	b	b	C	d
K	None	-	d	d	d	d

(3) Supplied by Acros Chemical with an $d_{3,2}$ particle size of

15 7.3 micrometres as measured on a Malvern Mastersizer.

(5) Kemilub ES supplied by Undesa

20

CLAIMS

1. A particulate detergent composition or component therefor which comprises a bi- or tri- valent metal ion carboxylic acid salt which, without the salt being present, has a hygroscopicity value of greater than 2%.
5
2. A composition or component as claimed in claim 1, wherein the metal ion is magnesium, calcium, zinc, aluminium, lead, iron or mixtures thereof.
10
3. A composition or component as claimed in claim 1 or claim 2, wherein the carboxylic acid salt has from 10 to 22 carbon atoms.
15
4. A composition or component as claimed in any preceding claim, wherein the carboxylic acid salt is magnesium stearate.
- 20 5. A composition or component as claimed in any preceding claim, wherein, without the carboxylic acid salt being present, has a hygroscopicity value of greater than 5%, preferably greater than 10%, more preferably greater than 20%, or even greater than 30%, or even greater than 40% or even greater than 50%.
25
6. A composition or component as claimed in any preceding claim, wherein the carboxylic acid salt is particulate.

7. A composition or component as claimed in claim 6,
wherein the carboxylic acid salt has a number average
particle size of from 0.1 to 500 micrometres,
preferably from 1 to 200 micrometres, more preferably
5 from 2 to 100 micrometres, most preferably from 3 to 50
micrometres, or even from 3 to 20 micrometres.
 8. A composition or component as claimed in any preceding
claim, wherein the carboxylic acid salt is present at a
10 level of from 0.1 to 10 wt%, preferably from 1 to 8
wt%, more preferably from 2 to 6 wt%.
 9. A composition or component as claimed in any preceding
claim, wherein carboxylic acid salt is present at the
15 surface of the particles at a higher concentration than
the average concentration in the composition or
component.
 10. A composition or component as claimed in claim 9,
20 wherein substantially all of the carboxylic acid salt
is present at the surface of the particles.
 11. A process comprising the step of adding a bi- or tri-
valent metal ion carboxylic acid salt to a previously
25 manufactured particulate detergent composition or
component.
 12. A process as claimed in claim 11, wherein the
carboxylic acid salt is particulate and is layered onto
30 the surface of the detergent composition or component.
-

ABSTRACT

5 A particulate detergent composition or component therefor
which comprises a bi- or tri- valent carboxylic acid salt,
preferably magnesium stearate, which, without the carboxylic
acid salt being present, has a hygroscopicity value of
greater than 2%.

PCT/EP2004/001512



This Page is inserted by IFW Indexing and Scanning
Operations and is not part of the Official Record

BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images include but are not limited to the items checked:

- ☒ BLACK BORDERS
- ☐ IMAGE CUT OFF AT TOP, BOTTOM OR SIDES
- ☐ FADED TEXT OR DRAWING
- ☒ BLURED OR ILLEGIBLE TEXT OR DRAWING
- ☒ SKEWED/SLANTED IMAGES
- ☐ COLORED OR BLACK AND WHITE PHOTOGRAPHS
- ☐ GRAY SCALE DOCUMENTS
- ☐ LINES OR MARKS ON ORIGINAL DOCUMENT
- ☒ REPERENCE(S) OR EXHIBIT(S) SUBMITTED ARE POOR QUALITY
- ☐ OTHER: _____

IMAGES ARE BEST AVAILABLE COPY.

**As rescanning documents *will not* correct images
problems checked, please do not report the
problems to the IFW Image Problem Mailbox**